Important Factors for Performing Percent Moisture Tests on Biological Matrices

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Abstract

Data for solid samples, including biological matrices, may be reported on a dry-weight or wet-weight basis. Sample results that are reported on a dry-weight basis account for the percent moisture in the sample by utilizing the percent moisture determined for that sample to correct the wet-weight analytical results. The decision to report sample results on a dry-weight or wet-weight basis is often determined by project or regulatory requirements and whether the end users need to compare sample results to risk-based standards or action levels that are developed on a dry-weight or wet-weight basis.

Most percent moisture procedures specify the use of several grams of biota sample to perform the test. Sample volume is typically not an issue when sampling and analyzing fish tissue, but would certainly become a question for the collection and analysis of mayfly or fish organs. Just how much sample volume is needed to perform a percent moisture test to provide the precision and accuracy required? Why would the precision and accuracy of the percent moisture determination be important? Following are data from percent moisture studies involving various sample amounts and time durations for which the test was performed.

Introduction

On December 22, 2008, an ash storage cell dike failure at the Tennessee Valley Authority (TVA) Kingston Fossil Plant in Kingston, Tennessee, resulted in the release of an estimated 5.4 million cubic yards of fly ash into the surrounding environment. As part of the comprehensive long-term monitoring program initiated following the release, TVA expanded its historical biological monitoring activities to include the sampling of a broad variety of biological specimens (*e.g.*, tree swallows, blue heron, osprey, benthic macro invertebrates, amphibians, turtles, and raccoons) for analysis of a suite of 26 ash-related metals constituents. These samples were analyzed for metals and were reported on a dry-weight basis based on sample-specific percent moisture. During data evaluation and risk assessment activities, observed variation in percent moisture values for a particular species resulted in an investigation to assess the factors contributing to moisture analysis.

The following items were suspected to contribute to the observed variation in moisture determinations:

- Limited mass available for percent moisture analyses of very small sample aliquots (*e.g.*, small bird eggs, certain amphibian species, or fish organs).
- Long-term frozen storage

Why is Percent Moisture Important?

- Analytical results are corrected for percent moisture to report data on a dry-weight basis.

  Dry-weight result = Wet-weight result / (1 - % moisture),
  where moisture is expressed as a decimal.

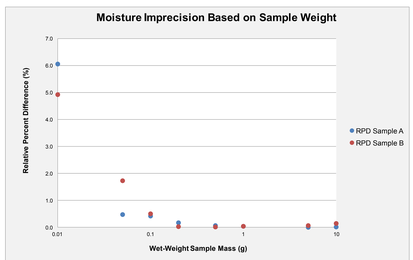
- Inaccurate or imprecise percent moisture results directly contribute to inaccuracy or imprecision of results for constituents of concern when the moisture results are used to convert concentrations to a dry-weight basis.

- Regulatory or risk-based standards may be specified on a dry-weight or wet-weight basis, requiring a % moisture determination to be performed to allow for the conversion of sample results between these bases.

Precision Evaluation for Small Sample Masses

The first part of the study focused on the precision of percent moisture determinations conducted using small sample masses. The laboratory determined the precision of percent moisture analyses performed using sample masses varying from 0.01 gram to 10 grams that were prepared (homogenized and sub-sampled) and analyzed on the same day.

Two separate samples of chicken egg contents were homogenized; percent moisture was determined in duplicate for each of these samples immediately (*e.g.*, within a few hours from homogenization).

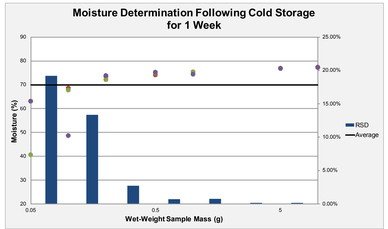


Relatively high variability was observed when moisture determinations were performed using very low initial wet-weight sample mass. As the initial wet-weight sample mass increased, the percent relative standard deviation approached zero.

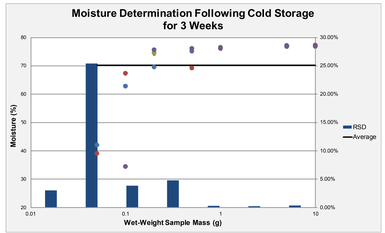
Effect of Long-Term Frozen Storage

In addition to minimum sample mass, frozen storage was suspected to contribute to percent moisture determination variability due to potential desiccation. The second part of the study focused on the impact of maintaining small sample masses in long term frozen storage.

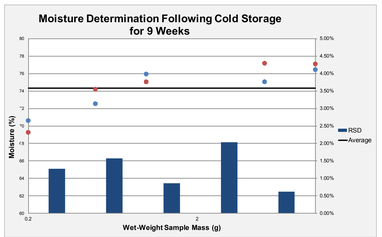
The sample aliquots measured in the first part of the study were subsequently held in frozen storage (< -10 °C) in wide-mouth jars for 1 week, for 3 weeks, and for 9 weeks.



Once frozen, the minimum sample mass necessary for acceptable precision for a percent moisture determination increased to 1 gram due to desiccation during frozen storage for up to 3 weeks.



Moisture determinations using initial sample wet-weights of greater than 2.3 g were observed to stabilize over longer-term storage (*e.g.*, 9 weeks frozen storage).



A second study was conducted using project samples with greater than 2.3 grams of wet-weight sample maintained in frozen storage demonstrated for up to 530 days. Acceptable accuracy and precision were demonstrated between these long-term measurements and the initially-reported percent moisture values for these samples.

Conclusions

Based on the results of the percent moisture study, it was apparent that sample mass and storage time both contribute to inaccuracy and imprecision in percent moisture determinations. The following conclusions were drawn from the study data.

- To achieve acceptable precision and accuracy, a minimum of 0.2 grams wet-weight sample mass is required when moisture determination is performed within 2 days from homogenization.
- To achieve acceptable precision and accuracy, a minimum of 1.0 gram of wet-weight sample mass is required when the moisture determination is performed within 3 weeks from homogenization.

Percent moisture determinations performed using greater than 2.3 grams wet-weight sample mass were demonstrated to be reproducible as long as 530 days from homogenization.

Recommendations

Percent moisture values are used as a correction factor in converted wet-weight basis results to dry-weight basis. As a result, any imprecision or inaccuracy in moisture determinations directly contributes to imprecision or inaccuracy of concentrations for constituents of concern when these are reported on a dry-weight basis. Accordingly, the following recommendations are offered.

- A minimum of 0.2 gram of sample must be collected for specific use in performing a percent moisture determination.
- Percent moisture determinations must be performed immediately following sample homogenization (*i.e.*, within 48 hours). If percent moisture determination cannot be performed within 48 hours from homogenization, a larger sample mass should be stored and used in the moisture determination.
- Applicable sampling Standard Operating Procedures and/or Sampling and Analysis Plans should stipulate the minimum mass sample collection requirement for percent moisture determination. Percent moisture determination should not be an afterthought.

Acknowledgement

The analytical work for this study was conducted by Pace Analytical Services, Inc. in Green Bay, Wisconsin.

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